

NATIONAL BUREAU OF STANDARDS

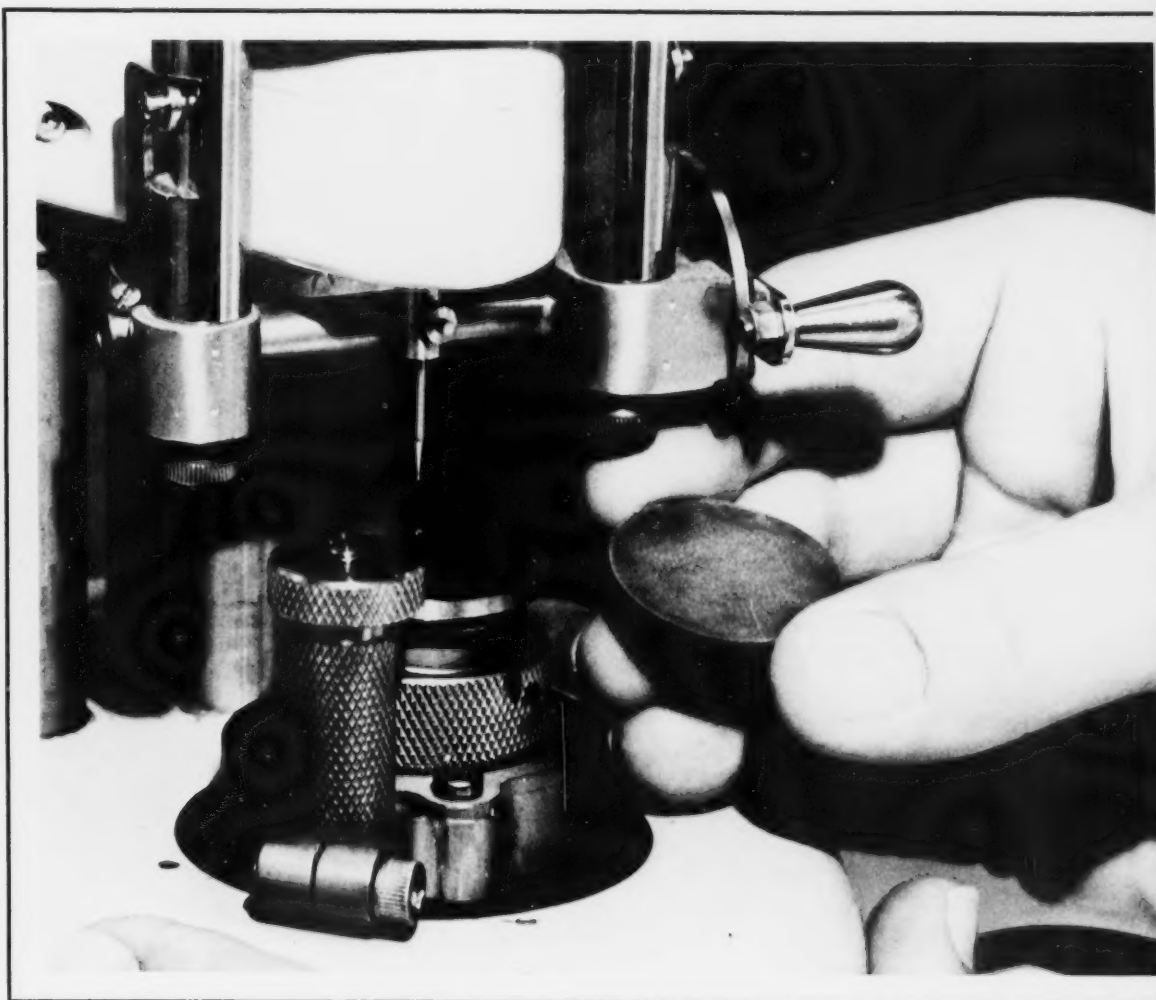
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NATIONAL BUREAU OF STANDARDS

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U.S. DEPARTMENT OF COMMERCE

Alexander B. Trowbridge
Secretary

NATIONAL BUREAU OF STANDARDS

A. V. Astin, Director

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COVER

This rubber specimen, which is about to be tested for hardness, contains phenyl beta-naphthylamine—a compound that greatly increases rubber stiffness. (See page 54.)

Prepared by the NBS Office of Technical Information and Publications, Washington, D.C. 20234

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The National Bureau of Standards serves as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. For this purpose, the Bureau is organized into three institutes—

- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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FLATTERY INDEX PROPOSED FOR ARTIFICIAL ILLUMINANTS

■ An aspect of artificial illumination that has received very little scientific study in the past is the amount of flattery it provides. Lighting installations for the home, office, factory, restaurant, or reception room are generally not intended for critical appraisal of colored objects. However, if a light-

ing installation for these purposes does flatter the people and objects viewed there, it will be preferred to one that is mercilessly revealing of flaws. For the same reason that cosmetics are used, a lighting system that enhances appearances performs a service. If, on the other hand, a store illuminates its meat with a light that flatters it, so that it appears redder, then ethical if not legal questions are raised. In either event, appraisal of illuminants for this characteristic has been needed.

As there has been no way in the past to appraise this aspect of illumination, D. B. Judd of the NBS Institute for Basic Standards has devised a method to evaluate the degree to which an artificial illuminant succeeds in flattering people and objects.¹ Although the proposed flattery index still requires some refinement, it appears to be a sound basis for further development. A practical index may make it possible for lamp manufacturers to develop artificial sources superior in flattery to daylight or to any of the conventional incandescent sources, and therefore capable of achieving greater public acceptance for appreciative viewing of selected colored objects.

The basis of the proposed flattery index is similar to the internationally-agreed-upon color-rendering index which evaluates the degree to which objects are rendered in their "true" colors. The color-rendering index for artificial daylight takes as the "true" color of an object the color in which it is rendered by the phase of natural daylight (direct sunlight, overcast-sky light, clear-sky light) intended to be duplicated by the artificial daylight. For example, a lamp is rated 100 if it renders a battery of test sample colors identical to those of the standard source. If the color of the sample differs under the source being appraised, however, the lamp is then rated at some value less than 100, depending on how large the difference is.

The proposed flattery index uses 10 of the 14 test samples selected for the color-rendering index; it uses the

continued

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D. B. Judd serves as a subject in a demonstration of the theory on which the NBS-proposed flattery index is based. A mercury-vapor lamp is the illuminating source.



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FLATTERY *continued*

same method to determine the reference or standard illuminant, and it uses the same scale to indicate degree of departure from the desired rendition. The essential difference is that the desired rendition is not the true color of the object, but the preferred color. It has been shown for example, that the preferred color for Caucasian complexions is redder and more saturated than it really is.² Similarly it has been shown that the color remembered for foliage is considerably less yellowish than the actual color.³

In appraising a light source, the proposed flattery index gives difference between actual and preferred complexion color about one-third of the total numerical value, difference between actual and preferred food colors (peas, lettuce, butter) about one-third of the total, and the remaining value is distributed among difference between actual and preferred colors of six other test samples. The preferred colors are taken to be more saturated than the actual colors.

Values of the proposed flattery index have been computed for various types of fluorescent lamps and one multivapor lamp. To obtain a degree of correspondence with the accepted preferences among these illuminants, it was found necessary to use only one-fifth of the difference between preferred and actual colors indicated in the literature, and even then the degree of correspondence, though encouraging, was found to be not altogether satisfactory. However, by making other choices of test samples and their preferred colors, and by choosing other percentages for their influence, it appears possible that a flattery index worthy of recommendation for practical use may be developed.

¹ For further details, see A flattery index for artificial illuminants, by D. B. Judd, *Illum. Engr.* 62, 593 (1967).

² Color preferences for natural objects, by C. L. Sanders, *Illum. Engr.* 54, 452 (July 1959); also, Color characteristics of human complexions, by G. B. Buck and H. C. Froelich, *Illum. Engr.* 43, 27 (Jan. 1948).

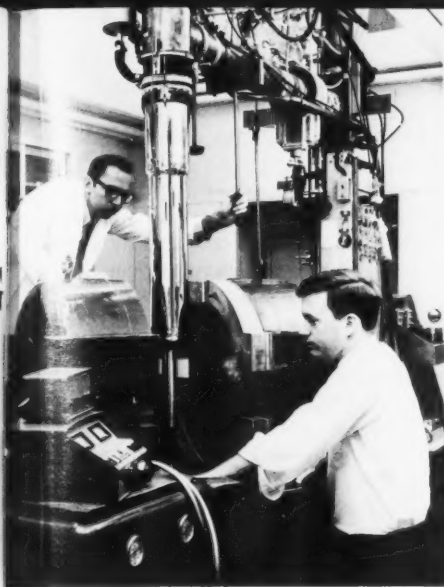
³ Memory colors of familiar objects, by C. J. Bartleson, *J. Opt. Soc. Am.* 50, 73 (1960).

SUPERCONDUCTING TRANSITION TEMPERATURES OF STRONTIUM TITANATE SEMICONDUCTOR

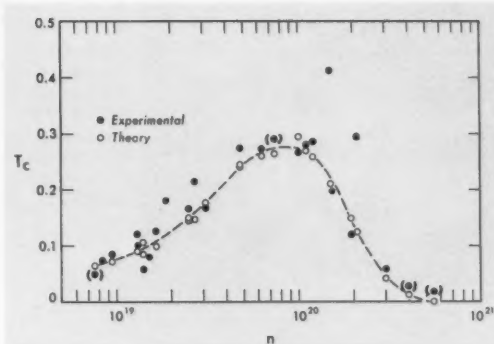
■ In recent experiments at the NBS Institute for Basic Standards, the superconducting properties of a material having very few conduction electrons were determined.¹ This was made possible by the previous discovery at NBS by J. F. Schooley that the oxide-type semiconductor, strontium titanate, is a superconductor.² Not only does superconductivity occur in strontium titanate with fewer conduction electrons than is the case for any other known superconductor, but the number of conduction electrons per unit volume can be varied over a very wide range (over three orders of magnitude) by sample preparation. This enabled J. F. Schooley and E. R. Pfeiffer of the Institute staff to measure the superconducting transition temperature and its dependence on the number of conduction electrons in SrTiO_3 .

Superconductivity—the phenomenon of a material losing all electrical resistance below a characteristic temperature called the superconducting transition temperature—has found increasing industrial uses in the last few years. Superconducting materials are used in efficient magnets, electron accelerators, cryogenic refrigeration apparatus, and digital computers. To apply superconductors to specific industrial applications, however, materials must be made which have the optimal superconducting properties for the intended use. For example, many industrial uses require superconductors having higher than average superconducting transition temperatures, to avoid the expense of working at extremely low temperatures.

To determine which materials are likely to have a given set of superconducting properties, a great deal of time and expense may be saved if it is possible to calculate the superconducting properties from the normal-state properties of a given material. The only alternative to this is to test for superconductivity.



E. R. Pfeiffer (left) and C. S. Koonce position a Dewar inside a magnet in preparation for an experiment to determine the superconducting transition temperature of strontium titanate as a function of conduction electron concentration.



Transition temperatures, T_c , as a function of carrier concentration, n , for superconductor strontium titanate show close agreement between experimental data (closed circles) and theoretical calculations (open circles).

Although the nature of superconductivity has been understood since 1957 when Bardeen, Cooper, and Schrieffer provided a theory of superconductivity,³ there have been few calculations of superconducting transition temperatures in specific materials. This is largely because the superconducting transition temperature is sensitive to many properties of the material, such as the effective mass of the electron, the interaction of electrons and lattice vibrations, and the average distance between electrons which is directly related to the conduction electron concentration. These properties in general are not known with sufficient accuracy to predict superconducting transition temperatures. In addition, for most superconductors, which are metals, none of the properties on which superconductivity depends may be varied independently.

For degenerate semiconducting strontium titanate, however, this is not the case, and the number of electrons per unit volume may be varied by proper preparation of the samples. A knowledge of how the superconducting transition temperature depends on a single parameter, the conduction electron concentration, allows scientists to assess the importance of the various normal state properties in determining superconducting properties.

In conjunction with the experimental measurements, therefore, the superconducting transition temperature as a function of electron concentration was calculated for strontium titanate by C. S. Koonce* and M. L. Cohen at the Berkeley campus of the University of California. When the experimental and theoretical curves were compared, it was found that by fixing a single normal-state parameter, the intervalley deformation potential, at one electronic concentration, the entire curve of transition temperature as a function of carrier concentration agreed quite closely with the experimental values. This agreement gives added

evidence that superconducting properties can be calculated from normal-state properties.

The strontium titanate crystals used in the investigation were prepared and the number of conduction electrons per unit volume was determined by W. R. Hosler of the Solid State Physics Section, NBS Institute for Materials Research. The crystals were cut to the desired shape from single crystal boules of pure strontium titanate. To introduce the desired carrier concentrations, the specimens were treated at temperatures ranging from 800 to 1400 K for periods ranging from 2 to 90 hours. Some samples were heated in flowing hydrogen gas and cooled under helium gas. Other specimens were sealed in evacuated quartz capsules with small known amounts of titanium, which acts as an oxygen getter. Still other samples were obtained from boules grown with appropriate amounts of niobium oxide powder added to the strontium titanate powder. All niobium treated samples were oxidized under oxygen gas at 700 to 800 kelvin for ten hours or longer.

The superconducting transition temperatures were obtained by measuring the magnetic susceptibility of each specimen from about 0.05 K through the transition temperatures using a mutual inductance apparatus. Below the superconducting transition temperature all lines of magnetic induction are excluded from the sample (the Meissner effect) resulting in large susceptibility. At the transition temperature, magnetic flux is no longer excluded and the susceptibility falls to its small, normal-state value.

¹ Superconducting transition temperature of semiconducting SrTiO_3 , by C. S. Koonce, M. L. Cohen, J. F. Schooley, W. R. Hosler, and E. R. Pfeiffer, *Phys. Rev.* **163**, No. 2, 380-390 (1967).

² J. F. Schooley, W. R. Hosler, and M. L. Cohen, *Phys. Rev. Letters* **12**, 474 (1964).

³ J. Bardeen, L. N. Cooper, and J. R. Schrieffer, *Phys. Rev.* **108**, 1175 (1957).

*Now at NBS.



H. H. Landon, Chief of the NBS Neutron Nuclear Physics Section, checks the NBS Reactor's core configuration through a periscope, while R. S. Carter, Chief of the Neutron Solid State Physics Section, watches.

NBS REACTOR ACHIEVES CRITICALITY

Health physicist H. deSpain inspects the top of the NBS Reactor for stray radiation between fuel loading steps.



■ On December 7, 1967, at 3:55 PM the National Bureau of Standards Reactor (NBSR) achieved criticality. A self-sustaining chain reaction was obtained after 20 fuel elements had been loaded into the reactor. Located at the Bureau's Gaithersburg laboratories, the facility will be operated at low power for testing purposes until funds become available for operation at its full power capacity of 10 megawatts.

This occasion was the culmination of more than nine years of planning and over four years of construction. NBS originated the conceptual design of the reactor and the architectural firm of Burns and Roe performed the engineering. Construction was carried out jointly by Blount Brothers, Inc. and NBS. Initial planning for the facility began in 1958 and construction was started in 1963 under an AEC license. The building was occupied in 1965 and the reactor completed late in 1967.

The NBSR was designed to provide NBS and other laboratories in the Washington area with an extensive central facility where neutron beams can be used for fundamental research on materials of all kinds. The primary need of these laboratories in a research reactor is for high-intensity thermal and subthermal neutrons. Such neutrons are used primarily to measure fundamental properties of ordinary matter, such as the location of atoms in a crystal or the forces between atoms. Knowledge of these basic properties of matter is needed to provide more precise standards for industry and new tools for research.

During the period between the loading of the first fuel element and full operation of the reactor, detailed testing must take place. Low-power tests are conducted with a partial load of fuel in the core; this positively limits the reaction. These tests are necessary to determine the characteristics of the reactor before increasing power for the next round of tests. Fuel is loaded into the core incrementally, increasing the neutron flux in the core each time until finally a "critical reaction" is achieved. At this point neutron multiplication in the core occurs rapidly enough to make the reaction self sustaining.

After criticality has been achieved, testing is continued to obtain additional information about the reactor before going to full power. The effect of fuel loading and core configuration on beam-tube flux must now be studied. In addition, precise maps of the flux in the core must be made under a variety of operating conditions so that accurate predictions of fuel burnup can be calculated and the power decay in the core determined. This phase must be completed, together with provision of adequate funds, before full scale operation can begin.

The change in neutron flux in the NBS Reactor is monitored in the control room as control rods are withdrawn during the fuel loading program.

NBS Technical News Bulletin

*Daniel Vomhof
adjusts the scale
of a recorder used
in the analysis
of corn syrups.*



CORN INDUSTRIES JOIN

RESEARCH ASSOCIATE PROGRAM

■ The Corn Industries Research Foundation (CIRF) has recently initiated a cooperative project with the NBS Institute for Materials Research under the Bureau's Research Associate Program. Daniel W. Vomhof, the CIRF's Research Associate at NBS, is working in the Institute's Analytical Chemistry Division to seek new methods of syrup analysis. Dr. Vomhof will put particular emphasis on developing a method of determining the dry solids content of syrups which will be both more accurate and more rapid than those in current use. The present methods depend on conversion tables developed many years ago for a limited range of syrup compositions that correlate dry solids content with (a) dextrose equivalence, and (b) refractive index measurements.

Corn refiners, like other manufacturers of chemical and related products, utilize many analytical methods in the course of conversion

of starch to its derivatives, including corn syrup. These methods are employed to control the quality of the syrup, to check the efficiency of production, and to provide customers with information on the composition of syrup. This information is therefore important to producers and users of corn syrup alike.

In recent years, development of new instruments and improvements in existing instruments have made possible many analytical techniques which have accuracy, precision, and speed not previously possible. The fact that new and improved production techniques have resulted in a greater variety of syrups and increasingly diversified applications makes improved analytical techniques essential to the corn refiners.

The three principal goals of the CIRF-NBS project are:

1. To evaluate several different analytical methods for the determination

of the percent water or, conversely, the percent dry solids in syrup in an attempt to find a method which is faster and more accurate than existing methods.

2. To determine the reliability of the new and existing methods when applied to the variety of syrups produced today and likely to be produced in the future.

3. To obtain a better understanding of the physical properties of syrups and the relationships between these properties, and to establish methods for their determination which could serve as the basis for standards for industries making and using corn syrups.

When these goals have been achieved, the CIRF hopes the results will be accepted by national and international organizations responsible for the standardization of products, product properties, and analytical procedures.

1967 STRATTON & ROSA AWARDS



John D. Hoffman (left) and Forrest K. Harris, 1967 recipients of the Stratton and Rosa Awards, respectively, hold the plaques presented them by A. V. Astin, Director (center).

■ On January 5, 1968, Dr. A. V. Astin, NBS Director, presented the 1967 Samuel Wesley Stratton Award to Dr. John D. Hoffman for "outstanding contributions in relating physical properties of solids to molecular phenomena and particularly with respect to chain folding in polymers"; and the Edward Bennett Rosa Award to Dr. Forrest K. Harris for "contributions to the development of standards for electrical measuring instruments." Both of these awards consist of a bronze plaque and a \$1500 honorarium.

The Stratton Award, founded in 1960 and first given in 1962, is named for the first Director of the National Bureau of Standards. In 1901, Dr. Samuel Wesley Stratton set about to organize NBS as a unique scientific institution. During his 21-year tenure of office, he firmly established the Bureau's position in the scientific and industrial community. Dr. Stratton left NBS in 1922 to become the ninth president of Massachusetts Institute of Technology. Each year, before the award is made, advisors from outside the Bureau are consulted in order to assure objectivity in the selection of the staff member or members whose achievements contribute most in support of the NBS mission.

Dr. Hoffman is Chief of the Bureau's Polymers Division. Since joining the Bureau in 1954, he has been concerned with theoretical work on dielectric relaxation in molecular crystals, dielectric properties of polymers, kinetics of crystallization in polymers, and precision dielectric measurements. In 1961, Dr. Hoffman was corecipient of an NBS Special Act or Service Award for an outstanding paper he co-authored on the crystallization of bulk polymers with chain folding. The Washington Acad-

emy of Science awarded him a certificate of recognition in 1962 "for distinguished service in the science of polymer physics."

The Rosa Award, first given for 1964, was named for Dr. Edward B. Rosa, a member of the Bureau's original staff and its first Chief Physicist. Throughout his career, Dr. Rosa was active in the development of standards of practice and made important original contributions of fundamental standards in the field of electricity and photometry. The Award gives recognition to outstanding accomplishments in the field of standards of practice—the standards by which industry judges its operations, its production processes, and the quality of its products. The technical and committee work on standards of practice are exceedingly demanding, requiring the highest degree of technical competence and administrative skill.

Dr. Harris is Chief of the Bureau's Absolute Electrical Measurements Section and was presented this Award specifically for his work as Chairman of an American Standards Association (now United States of America Standards Institute) committee which completely revised the American Standard Code for Electrical Metering. This widely used and very important code governs the specifications and performance of the many electrical watt-hour meters in American homes and businesses. In 1955, Dr. Harris received the Department of Commerce Meritorious Service Award for his professional accomplishments in the field of electrical instruments and measuring services, and for authorship of the book, *Electrical Measurements*.¹

¹ *Electrical Measurements*, by Forrest K. Harris, John Wiley and Sons, Inc., New York, N.Y., 1952, \$8.

BIRMINGHAM APPOINTED EXECUTIVE OFFICER AT BOULDER

■ NBS Director A. V. Astin recently appointed Bascom W. Birmingham as the Executive Officer of the NBS Boulder Laboratories, Boulder, Colo. While assuming the broader duties of Executive Officer, Mr. Birmingham will continue as Chief of the Cryogenics Division. This division is the

principal organization in the Federal Government for providing comprehensive and current information in the field of very low temperatures.

As Executive Officer, he will plan and supervise the administrative programs required to support the Bureau's scientific and technical programs at Boulder, and will act as the personal representative of the Director in all matters at Boulder requiring a spokesman.

Mr. Birmingham joined the Bureau staff in 1951. As a part of his early work he assisted with the installation of the large-capacity hydrogen liquefying equipment at Boulder. He later served as Chief of the Cryogenic Equipment Section and in this capacity was responsible for the design of experimental vessels for the storage

and transportation of liquefied gases, including suitable refrigeration equipment. In 1955, he represented NBS as consultant and advisor to the University of California Lawrence Radiation Laboratory at Berkeley on the design of a large liquid-hydrogen bubble chamber that was later used in conjunction with the bevatron high-energy particle accelerator.

The author or coauthor of some twenty technical papers, he is also the coauthor of a chapter on liquid hydrogen bubble chambers in the book *Technology and Uses of Liquid Hydrogen*, published in 1964 by Pergamon Press, Ltd., Oxford, England. Recently, he was named American Editor of *Cryogenics*, an international journal of low temperature engineering and research.

MODEL DEVELOPED FOR ESTIMATING COOLDOWN TIME OF CRYOGENIC SYSTEMS

■ The broad scope of cryogenic applications in such areas as steel manufacturing, aerospace engineering, natural gas liquefaction, and frozen food industry necessitates the storage, transportation, and transfer of large volumes of cryogenic liquids. Difficulties exist, however, particularly in the transfer process, because uncooled pipelines are generally used to convey a cryogenic fluid from one point to another. Hence, the lines must first be cooled from ambient temperature to near the liquid's boiling temperature. During most of this cooldown period, which for large systems may take many hours, the liquid boils and the pipe delivers only warm gas. Thus, the time that elapses before the pipe starts delivering liquid can be an important operational consideration.

Recently a "flow-controlled model" was developed by W. G. Steward, R. V. Smith, and J. A. Brennan of the NBS Institute for Materials Research at the Cryogenics Laboratory, Boulder, Colo. Developed under sponsorship of the National Aeronautics and Space Administration, the model offers a quick and easy method for estimating cooldown time for simple* cryogenic pipelines of relatively large length-to-diameter ratio. It is expected to be of value

in the design of cryogenic pipelines, in the fueling of rockets, and in the storage and transfer of cryogenic liquids for commercial uses.

The model is basically a system of mathematical equations relating the parameters involved with flow. Through the use of the flow model, the cooldown time can be estimated from a dimensionless parameter read from a graph. It is only necessary to know the fluid and pipe enthalpy along with the density and velocity of sound in the warm gas to utilize this method.

The flow model was developed largely from observation of the experimental cooldown data obtained from an experimental system, designed and constructed at NBS, and from reported observations in the literature. The experimental system consists basically of a supply Dewar with a 200-ft. long, 0.625-in. I.D., 0.75-in. O.D., vacuum-insulated, copper transfer line, discharging to the atmosphere. Tests were also conducted with the line shortened to 150, 82, and 25 feet.

Experimental results show that in simple piping systems with relatively large length-to-diameter ratio, the most important factor controlling the cooldown is the resistance

continued

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CRYOGENIC *continued*

to flow of vaporized liquids. Comparison of the experimental system with the model showed the cooldown process of simple systems approaches that of the proposed NBS model quite closely.

For complicated systems, pipelines with small length-to-diameter ratios, systems with appreciable heat leak, constrictions, or concentrated masses of metal, a more com-

prehensive model is needed. Investigation is currently underway in the NBS Cryogenics Division to produce such a model capable of giving a complete cooldown history along a pipeline, including such factors as pressure surging, flow reversals, and flow rates.

²A simple system is defined here as one which has a constant flow area, is without concentrated masses, orifices or other constrictions, and is well insulated.

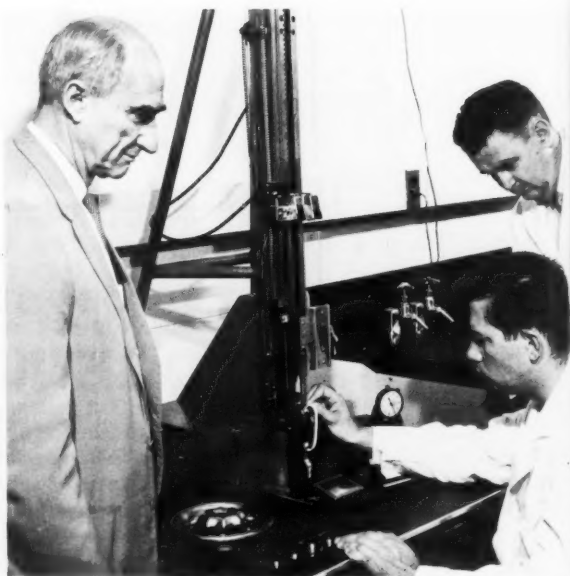
R. D. Stiehler, E. J. Parks, and F. J. Linnig prepare a specimen of rubber reinforced with phenyl beta-naphthylamine for a strain test.

NATURE OF RUBBER REINFORCEMENT

■ A long-term study at NBS promises to shed new light on the nature of rubber reinforcement. This study has shown that certain crystalline organic compounds containing a beta-naphthyl group produce an increase in the stiffness or reinforcement of vulcanized rubber.¹

Such reinforcement is normally obtained by mixing a reinforcing filler into the rubber before vulcanization. While the exact mechanism is unknown, there are two theories concerning the nature of rubber reinforcement with materials such as carbon black. One hypothesis is that chemical bonds exist between the rubber and the filler; the other is that reinforcement is a physical phenomenon only.

About 5 years ago NBS chemists unexpectedly found a remarkable stiffener of elastomers that strongly supports



the hypothesis that physical forces alone can produce a profound reinforcement of rubber. Investigators Frederic J. Linnig, Edwin J. Parks, and Robert D. Stiehler² of the Bureau's Institute for Applied Technology were then studying the effect of zinc and lead oleates on the stiffness of lightly vulcanized natural rubber. Inadvertently, an antioxidant, phenyl β -naphthylamine (PBNA), had been introduced in a larger amount than had been intended. A profound stiffening resulted which was later found to be caused by the PBNA.

Subsequent investigations showed that when crystalline compounds containing a β -naphthyl group were introduced into vulcanized rubber under certain conditions, substantial reinforcement of the rubber resulted. These compounds include phenyl β -naphthylamine (PBNA),

naphthol, phenyl β -naphthylene, and β -naphthylamine. Of these compounds PBNA produced the most profound reinforcement. About 4 percent PBNA under certain conditions is comparable to about 25 percent carbon black in stiffening rubber.

The astonishing thing was that the PBNA could be removed merely by extraction with benzene and, after drying, the rubber returned to its original degree of stiffness. Although this reversible nature of reinforcement with PBNA strongly indicates a physical mechanism, the stiffening cannot be explained by existing theories of physical reinforcement.

Attempts to obtain this stiffening with unvulcanized rubber have not been successful, but with vulcanized rubber the results showed that the type of vulcanization was not a factor in the stiffening of the rubber. Sulfur vulcanization, sulfur-accelerator-zinc oxide vulcanization, and sulfurless vulcanization, such as those employing tetramethylthiuram disulfide or peroxide, all show about the same stiffening with PBNA.

The effect of PBNA with various elastomers was investigated using two natural rubbers (Liberian crepe and smoked sheet) and three synthetic rubbers (*cis*-polyisoprene (IR), styrene-butadiene (SBR), and butyl (IIR)). The results showed that PBNA caused a very pronounced stiffening of the two natural rubbers and SBR, and a pronounced but lesser stiffening in IR and IIR. The reason for the smaller amount of stiffening with the latter two rubbers was not investigated. Nevertheless, it is apparent that the stiffening effect is a general phenomenon and is not restricted to a specific type of elastomer.

Most of the tests were made on vulcanized strips 1 or 2 mm in thickness and 150 mm long by 9 mm wide. The PBNA was introduced by swelling the vulcanized strips in a benzene solution containing the desired quantity of PBNA to be tested. The specimens were then dried under a vacuum at room temperature. From the swelling volume ratios and the concentration of the PBNA in the benzene solutions, the percent of PBNA in the dried specimens was calculated.

Measurements of strain after 1 minute periods under a load of 5 kg/cm² were made on the strips according to ASTM designation D1456-61.³ Young's modulus was calculated from the strain data using the relationship of Martin, Roth, and Stiehler.⁴ Young's modulus was also determined from measurements of hardness made with a microtester in accordance with ASTM designation D1415-62T.⁵ Strain is a measure of modulus in the lengthwise direction and hardness is chiefly related to modulus in the thickness direction.

The PBNA had little effect below 2 percent, but between 2 and 4 percent there was a precipitous increase in Young's modulus for both strain and hardness. Above 4 percent PBNA strain measurements, there was a continued increase in modulus, whereas hardness measurements showed only a slight increase from 4 to 18 percent PBNA.

Thus the stiffening at the higher concentrations of PBNA was not isotropic.

The effect of temperature on PBNA stiffening of vulcanizates was also examined. When PBNA is absent, the strain values tend to decrease when the temperature increases, as expected. On the other hand, the strain values of specimens containing PBNA increase markedly as the temperature increases. The negative thermal coefficient for the modulus of PBNA-reinforced vulcanizates is similar to that observed for carbon black vulcanizates. Further experiments involving PBNA solubility in rubber at various temperatures are necessary to establish an accurate thermal coefficient since it was observed that stiffening was associated with crystalline PBNA and not with PBNA dissolved in the rubber.

Measurements on creep (continued elongation with sustained tension) and residual set (percentage by which specimen fails to return to its original dimensions when tension is removed) were conducted over a range of conditions. Creep is less pronounced with specimens containing PBNA, and decreases with increased PBNA content. Residual set, after recovery at 24 °C, first decreases and then increases again with increased PBNA content. This large residual set after recovery at room temperature for specimens containing an increased PBNA content may be caused by partial solution and reformation of PBNA crystals which restrict recovery. This restraint is apparently removed when the specimen is heated to 70 or 100 °C during recovery.

The extent of breakdown on cycling was determined with a tensile tester. Stress at 100 percent elongation was measured after each extension. Repeated stretching causes a much greater reduction in stress for elastomers stiffened with PBNA than for elastomers stiffened with carbon black. Although considerable breakdown does occur on cycling the PBNA specimens, an appreciable portion of the stiffening still remains.

This investigation suggests that reinforcement of rubber is a physical phenomenon determined by the surface area of the filler and the interfacial forces per unit area between the filler and the rubber. Interfacial forces are maximized when the molecular or atomic structure on the filler surface is similar geometrically to the molecular structure of the rubber and when there is sufficient freedom of motion to align the atoms to permit attraction in the filler and the rubber at all or most possible points.

³ Stiffening of elastomers by organic fillers, by R. D. Stiehler, E. J. Parks, and F. J. Linnig, *Polymer Preprints* 8, No. 2, 1563 (Sept. 1967).

⁴ Effect of certain crystalline substances on physical properties of elastomers, by F. J. Linnig, E. J. Parks, and R. D. Stiehler, *Rubber Chem. Technol.* 39, 1041 (1966).

⁵ ASTM Standards on Rubber Products (with Related Information), 20th ed., Committee D-11 on Rubber and Rubberlike Materials, American Society for Testing and Materials, Philadelphia, Pa., p. 664, 1961.

⁶ Behavior of pure gum rubber vulcanizates in tension, by G. M. Martin, F. L. Roth, and R. D. Stiehler, *Trans. Inst. Rubber Ind.* 32, 189 (1956); *Rubber Chem. Technol.* 30, 876 (1957).

⁷ 1962 Supplement to Book of ASTM Standards including Tentatives, Part II, Rubber, Electrical Insulation, American Society for Testing and Materials, Philadelphia, Pa., p. 72, 1962.

■ Radio signals within circuitry and to and from antennas are often carried by a coaxial transmission line, which consists of a center conductor surrounded by another conductor in the form of encircling tubing. The outer conductor acts as a shield; thus coaxial transmission lines pick up little interference from adjacent fields and conduct signals with little loss.

The spacing of the two conductors and the dielectric constant of whatever lies between them determine the impedance of the coaxial transmission line, which should be constant over its entire length. The desired spacing is maintained in a rigid coaxial line by insulating supports, which are placed at intervals down the line. Since the

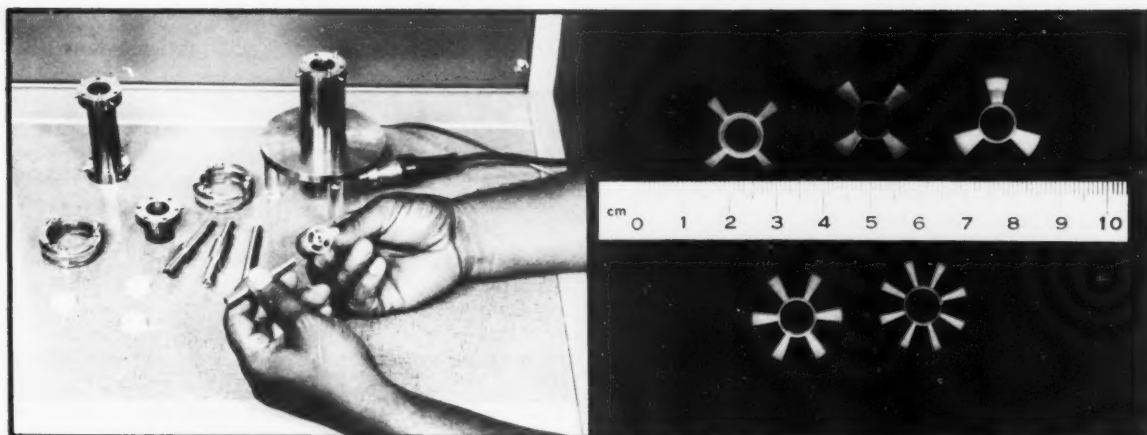
materials used all have dielectric constants greater than that of air (the dielectric in the spaces between supports), the impedance would change at the supports if the ratio of the conductor diameters were not changed at the support in compensation. These abrupt changes in diameter cause "step discontinuities"; although necessary for constancy of the characteristic impedance, they unfortunately produce reflections in the conductor.

The effects of step discontinuities can be reduced by cutting away the dielectric support to as small a volume as possible, so that less reduction of the conductor diameter is necessary. Until recently, however, there was no way to find the effective dielectric

constant resulting when the support was cut back, so the compensatory undercutting required could only be estimated. Now an equation giving the effective dielectric constant of multielectric coaxial sections has been developed at the NBS Radio Standards Laboratory, Boulder, Colo., by J. E. Cruz, enabling the radio engineer to determine the exact undercutting necessary.

Mr. Cruz validated the equation by producing supports having different configurations but, according to the equation, the same effective dielectric constant. When tested in transmission lines their dielectric constants were found to be virtually identical. The experimental results demonstrate that,

COAXIAL TRANSMISSION LINES WITH MINIMAL DISCONTINUITIES



A newly designed support is placed on the center conductor of a test section of a rigid coaxial transmission line. (Parts of outer conductor stand at rear.) Supports of alternative designs, at lower left, have dielectric constants computed to be the same.

These five insulating supports for rigid coaxial transmission lines have different configurations but the same effective dielectric constant.

the use of the equation described, an approximate value can be found for the effective dielectric constant of center conductor supports in coaxial transmission lines. This information is invaluable in determining the best possible support configuration prior to construction.

Effective Dielectric Constant

The formula giving the effective dielectric constant of a multidielectric support was obtained by applying Gauss' Law for the voltage between two coaxial cylinders to obtain the capacity per unit length of coaxial line as a function of the dielectric constant. A formula giving the effective dielectric constant of a two-

dielectric system was obtained by use of the definition of characteristic impedance:

$$\epsilon' = \frac{\ln \frac{d}{a'}}{\frac{1}{\epsilon_1} \ln \frac{d}{a'} + \left(\frac{1}{\eta} - \frac{1}{\epsilon_1} \right) \ln \frac{d}{b'}}$$

Here ϵ' is the effective uniform dielectric constant and ϵ_1 that of the support material, a' is the cut-down diameter of the center conductor, d the outer diameter of the support, b the inside diameter of the cutaway portion of the support, and η a factor determined by the arc length of the radial support sections.

The equation is based on the assumptions that the length of the di-

electric support is infinite and that all electric fields in the support are in the radial directions. To meet these assumptions it must be assumed further that there is no interaction between the fringing field due to the step discontinuity and the field in the support.

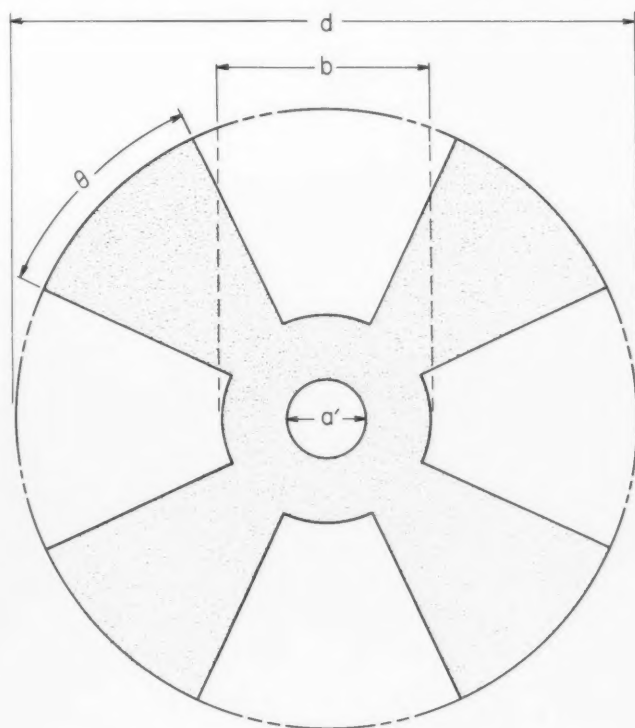
Cutaway Line Support

The Bureau also experimented with different configurations of cutaway transmission line supports. The experimental supports were made from rexolite disks, which were milled out from the side on radii in a symmetrical pattern. Configurations having three and more "legs" were made; the five-legged one, for instance, looked somewhat like a starfish with legs joined to its body at their narrow ends.

Experimental Verification

To check the validity of the support configuration formula, Mr. Cruz measured the capacitance change of a dielectric support in a guarded coaxial transmission line for changes in the arc length of the radial members of the support. First, capacitance measurements were made of a disk of the support dielectric sequentially as material was removed from the radial members of the support; the plot of values agreed with predictions from the formula. Then for a fixed number of the radial members, material was removed closer to the center in steps; here, too, the values agreed with plots from the formula.

To further illustrate the validity of the equation, five additional dielectric supports with different radial geometries were fabricated to the same effective dielectric constant by using the equation. Four of the five supports deviated less than 1 percent from the projected effective dielectric constant. The remaining support deviated less than 1.5 percent.



DIELECTRIC CONSTANT OF AIR ϵ_2
 SOLID ϵ_1

TWO-DIELECTRIC SUPPORT

Configuration of a support for a rigid coaxial transmission line, consisting of four radial elements of θ radians each and a diameter d , with intermediate sectors cut out to a diameter b .



NEWS

This column regularly reports significant developments in the program of the National Standard Reference Data System. The NSRDS was established in 1963 by the President's Office of Science and Technology to make critically evaluated data in the physical sciences available to science and technology on a national basis. The System is administered and coordinated by the National Bureau of Standards through the NBS Office of Standard Reference Data, located in the Administration Building at the NBS Gaithersburg Laboratories.

High-Quality Spectral Data

Since the NSRDS was set up in 1963, advisors to the System have agreed with users of data that molecular spectral data projects (infrared, ultraviolet, NMR, mass spectra) should be given very high priority. In making plans for such projects, however, the Office of Standard Reference Data has had to consider two factors—first, there is a great diversity of uses for molecular spectral data (ranging from automatic monitoring of chemical industrial processes to statistical mechanical calculations) and their quality demands are equally diverse; and second, there are several already-active suppliers of spectral data, some of whom depend on the sale of spectra to keep their activity going.

One early NSRDS project was established to provide additional infrared spectra of moderate quality. It has now become apparent that this project will indeed result in a useful collection of spectra, but that its continuation as part of the NSRDS may not be justified on economic grounds. In fact, there are indications that newly-measured high-quality infrared spectra would cost less per unit than the spectra now being collected.

Both industrial and academic users of infrared spectra have expressed a definite interest in the availability of a small collection of high-quality Class II infrared spectra¹ which could serve several purposes:

- (a) A benchmark, or standard, for the capabilities of present-day instruments;
- (b) a challenge to professional spectroscopists which could result in substantial upgrading of laboratory spectroscopy in general;
- (c) a foundation for advances into still-higher quality spectroscopy;
- (d) a test case to determine the suitability of high-quality spectra in solving generally-encountered problems of chemical analysis;
- (e) a potentially valuable educational tool;
- (f) a pilot study to indicate the absolute cost and the cost-benefit ratio of high-quality spectra.

The Office of Standard Reference Data is now working to set up a pilot-level Class II infrared spectral data project in which several private laboratories could participate.

The same considerations of quality level and user needs apply to other branches of molecular spectroscopy; and an NSRDS study is now underway to formulate a statement of quality standards and needs in NMR spectroscopy. The benefits which may be realized should be the same as in infrared spectroscopy. One of the major advantages of increased emphasis on high-quality spectral data is the opportunity to maintain the traditional NBS role of setting standards, while encouraging the participation of the entire interested scientific community.

Atomic Spectra—Silicon I

Recent publication² of NSRDS-NBS-3, Section 2, *Selected Tables of Atomic Spectra, Atomic Energy Levels and Multiplet Tables, Si I* (20 cents), provides a second step in the current NBS revision of data on atomic spectra as derived from analysis of optical spectra. These data were originally presented in two sets of tables, both by Charlotte E. Moore. *Atomic Energy Levels*, NBS Circular 467, consisted of three volumes published in 1949, 1952, and 1958, while *An Ultraviolet Multiplet Table*,

NBS Circular 488, was published from 1950 to 1962.

The present series, also by Dr. Moore, combines the two types of data. Separate sections on individual species are published as they are completed, so that the material may be made available to users as rapidly as possible.

NSRDS-NBS-3, Section 1, covered Si II, Si III, and Si IV. Section 2 covers Si I. With this work Dr. Moore is continuing her outstanding contribution to the quantitative information resources of physics in general, and especially astrophysics, plasma physics, and space technology.

Compendium of *ab initio* Calculations of Molecular Energies and Properties

Morris Krauss, of the NBS Division of Physical Chemistry, has recently completed a review and resource analysis entitled *Compendium of ab initio Calculations of Molecular Energies and Properties*, NBS Technical Note 438² (70 cents). The review was requested and supported by the Advanced Research Projects Agency of the Department of Defense, and is part of the National Standard Reference Data System program in Atomic and Molecular Properties. Both users and students interested in quantum mechanical calculations have found it increasingly difficult to determine the present status of these calculations. This compendium references the work from 1960 to the present and abstracts from the mass of data the best values for several observable properties, including the total energy, dissociation energy, electron affinity, spectroscopic constants, electric moments, field gradients, polarizabilities, and magnetic constants. To provide an insight into molecular electronic structure, tables of orbital energies are also included.

This compendium directs attention to the capabilities and deficiencies of quantum mechanical calculations by presenting the best results in a reasonably compact form. Its usefulness will undoubtedly be limited in time by rapid advances in the field. Nevertheless, it provides a guide and summary of resources which will be of great assistance to workers in the field. It should also aid in planning future calculation projects by indicating clearly the present state-of-the-art.

Tables of Bimolecular Gas Phase Reactions

Tables of bimolecular gas phase reactions have been compiled by A. F. Trotman-Dickenson, of the University of Wales, Aberystwyth, and his associate, G. S. Milne of Brandeis University, Mass. Published as NSRDS-NBS-9² (\$2), this work is the first product of the National Standard Reference Data System program in Chemical Kinetics.

The survey covers the kinetics of bimolecular and termolecular gas reactions not involving electronically excited states for data published from 1954 through 1965. While the authors originally hoped to make a critical study and to provide recommendations of reliable data in most cases,

they found that the information in the literature would not justify this approach. They therefore attempted to make the tables as comprehensive as possible. Footnotes to the tables provide indications of reliability where appropriate, as well as other comments on the significance of the numerical values and on the techniques employed.

These tables should be useful to all workers in gas phase kinetics. The systematic presentation in compact tabular form of the large volume of data in this area will provide many chemists and engineers with the numerical values they need. Also, the references to the original literature sources will give the specialist easy access to further details.

Local-Currency Funds Used to Aid Data Compilation Projects

A project initiated by the Office of Standard Reference Data in September highlights some of the complexities of the data compilation and evaluation process and shows the advantages of international cooperation in such activities. The Chemical Thermodynamics Data Group at NBS compiles and evaluates data on the heats and entropies of formation of chemical compounds. Since there is a growing experimental effort in this area in the Soviet Union and the Eastern European states, the literature from these sources is increasing in scope and importance. Although some members of this Data Group have learned to read Russian, efficient, rapid extraction of information from these sources requires a bilingual fluency they have not attained. However, there are many scientists in Israel who are fluent in English and various Eastern European languages, and some of them are employed by the Israeli Translation Service, which, in turn, has a contract for abstracting and translation with the National Science Foundation using local-currency funds accruing to the United States from the sale of agricultural commodities. Thus, the Chemical Thermodynamics Data Group is attempting to cover the Eastern European literature with the help of the Israelis through the NSF contract.

Thermodynamic and Transport Properties of Steam Published by ASME*

The American Society of Mechanical Engineers has recently published a new set of tables and charts for the thermodynamic and transport properties of steam. This is more than just another set of thermodynamic tables. It is an important milestone in an interesting data evaluation project involving industrial, educational, and governmental cooperation both in this country and abroad. This cooperation has extended over half a century starting with the establishment in 1921 of the ASME Steam Tables Committee.

*The 1967 ASME Steam Tables are reported here in accordance with the policy of the Office of Standard Reference Data to announce to the technical public new data compilations having the status of standard reference data. The fact that these tables are in the English system of units, rather than the International System of Units (SI), reflects current American engineering practice. The German Engineering Society will shortly issue steam tables in SI units that are consistent with these.

NSRDS NEWS *continued*

The Thermodynamic and Transport Properties of Steam comprise nine tables and thirteen charts on steam and water. The data were calculated from the equations adopted by the International Formulation Committee and published in its 1967 *IFC Formulation for Industrial Use*. This volume³ was prepared by C. A. Meyer, R. B. McClintock, G. J. Silvestri, and R. C. Spencer, Jr.

The long history of international cooperation leading up to these tables is of particular interest today when such cooperation, especially in the area of standard reference data, is being increasingly emphasized. For this reason, the following historical account, much of which is taken from the introduction to the publication, is presented.

In the 1920's, the ASME Steam Tables Committee took advantage of parallel interests in England, Germany, and Czechoslovakia to achieve a coordinated research program of international scope, and, moreover, to provide a formal machinery for reaching international agreement on the "best values" for the properties of this important substance. Such international agreement was important even then in providing a uniform standard for the specification and performance of equipment for generating and utilizing steam.

The First International Conference on Properties of Steam (London 1929) concerned itself with a detailed examination of the tables of specific volume and enthalpy submitted by the various national delegations. The skeleton table compiled at the conference served as the basis for the 1930 ASME Steam Tables (prepared by J. H. Keenan and published by ASME). This conference recognized that considerable additional measurements would be required before a truly satisfactory representation of these properties could be agreed upon.

Progress was reviewed at the Second International Conference (Berlin 1930), at which time significant modifications were made in the skeleton table. The time, however, was not ripe for full agreement. Such agreement was reached in 1934 at the Third International Conference held at NBS in Washington, at Cambridge, and in New York with the adoption of a skeleton table giving the specific volume and enthalpy along the saturation line and at round values of temperature and pressure for the compressed liquid and superheated vapor. In addition, each entry in the table had associated with it a tolerance, so that the two values would "constitute a criterion, internationally agreed upon, by which the reliability of a steam table may be judged." The skeleton table and the experimental measurements which led up to it served as the basis for a number of publications, the most prominent of which was the Keenan and Keyes Steam Tables which has served as the handbook of engineers these past 30 years.

The need for continued research on properties of steam and water was recognized by the Third International Conference, to the extent that solicitations and even recorded

assurances from various delegations and institutions guaranteed that the work on steam would be continued and extended.

The plan to hold a Fourth International Conference in Prague, Czechoslovakia, however, gave way to World War II. Almost twenty years elapsed before the thermodynamic properties of steam again became an active concern of ASME and the international scientific and engineering community. When the Fourth International Conference was held in Philadelphia in 1954, it was evident that technological advances made in the intervening years would require thermodynamic information outside of the temperature and pressure range of the existing steam tables. ASME established a Research Committee on the Properties of Steam, and charged it with the task of arranging for new research and of re-establishing the program of international coordination which had proved so fruitful two decades earlier. ASME also accepted the invitation to serve as the permanent Secretariat for the International Steam Conferences.

By this time, in Japan and the U.S.S.R. interest had developed in thermodynamic tables for steam having the status of an international standard. Moreover, a considerable body of new experimental measurements had been reported or started by a number of institutions in the Soviet Union. Thus, at the time of the Fifth International Conference (London 1956) it was evident that the work of examining the published data and supervising the then current research could be handled best by national commissions from the four countries in which the major experimental research was being carried on—the Federal Republic of Germany, the United Kingdom, the Union of Soviet Socialist Republics, and the United States of America. It was further agreed that the national commissions would constitute the International Coordination Committee—the working group of the Fifth International Conference.

The labors of the International Coordinating Committee were rewarded at a plenary session of the Sixth International Conference (New York 1963) when the table was adopted as "The International Skeleton Table of 1963." The temperature range of the skeleton table is from 0 to 800 °C and the pressure extends to 1000 bars—a considerable extension over the range of the 1934 Skeleton Table. Where new experimental data reflected refined experimental techniques, the "tolerance" limits of the new table narrowed. The basis for the equations from which these latest ASME Steam Tables have been computed is the skeleton table adopted by 16 member nations of the Sixth International Conference.

¹ NSRDS News, NBS Tech. News Bull. 50, No. 9, 166 (Sept. 1966).

² Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for the price indicated.

³ Available from the American Society of Mechanical Engineers, 345 East 47th St., New York, N.Y. 10017, for \$12.50.

ACCURATE PHASE ANGLE MEASUREMENTS AT AUDIOFREQUENCIES

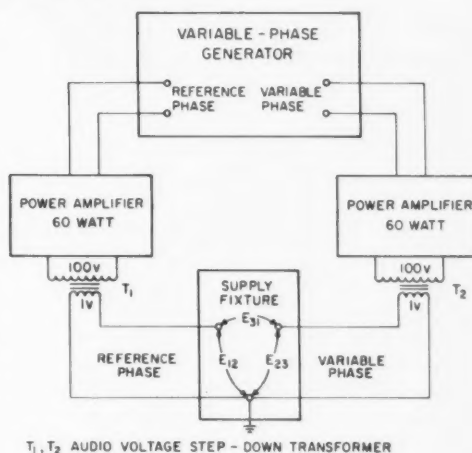
A method of accurately determining the phase angle of audiofrequency voltages has been developed¹ by W. W. Scott, Jr., of the NBS Radio Standards Laboratory at Boulder, Colo. The procedure uses equipment normally found in the standards laboratory, which has been assembled to measure phase angle differences from 0° to 360° for frequencies from 10 Hz to 10 kHz.

By this method, measurements between 0° and 170° and between 190° and 360° are made with uncertainties of $\pm 0.05^\circ$ to $\pm 0.01^\circ$, and measurement of an angle of 60° can be repeated within a range of 0.001°. Performance is relatively unaffected by the presence in the input signals of harmonics, which can degrade the accuracy of phase measurements made by other methods. Ultimately this development should be useful in laboratories calibrating phase angle standards, perhaps over a range extended to 100 kHz.

The procedure is based on a "three-voltmeter method." Usually, in this method, the phase angle between two alternating voltages is determined by measuring the magnitude of three voltages between terminals of the source. However, by using thermal voltage converters and appropriate inductive voltage dividers, the ratios of these voltages can be more conveniently determined and the phase angle can then be calculated by using the law of cosines.

The supply system is an important part of the phase-angle standard. A supply fixture provides convenient physical terminals to which voltages can be supplied, at which voltage comparisons can be made, and to which phase-angle standards can be connected for calibration. The voltage level for each phase is most conveniently controlled by a potentiometer (voltage divider) connected between

Equipment components and connections used in the NBS-developed method for accurately measuring audiofrequency phase-shifts. Shift is determined from three measurements at supply fixture.



T₁, T₂ AUDIO VOLTAGE STEP - DOWN TRANSFORMER

the voltage generator and a power amplifier. A variable-phase generator indicates the approximate phase difference between the reference and variable phases so that any ambiguity in angle can be resolved.

The detector system is another important part of the phase-angle standard. This system consists of two thermal voltage converters, a Lindeck potentiometer, and a galvanometer.

In order to measure phase angles from $\pm 6^\circ$ to $\pm 60^\circ$, a decade resistance box, an a-c detector, and an inductive voltage divider also must be included in the circuit. The phase angle is found by use of a prescribed procedure in which the value obtained from the inductive voltage divider is substituted in a trigonometric formula.

For phase angles between 0° and $\pm 6^\circ$, an a-c differential voltmeter replaces the decade resistance box and the a-c detector in the circuit.

Phase angles between $\pm 60^\circ$ and $\pm 170^\circ$ are measured using a different arrangement of the apparatus used for the $\pm 6^\circ$ to $\pm 60^\circ$ measurements and using the reading of the inductive voltage divider in a different trigonometric equation. Still another method

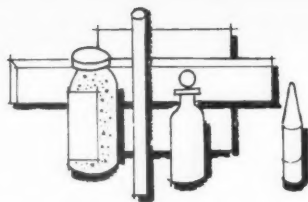
is required for good precision for phase angles of 170° to 190°.

The standard can be used with sinusoidal or slightly distorted sinusoidal waveforms. If the maximum magnitudes of the harmonics in the two power supplies are equal, measurement error due to nonsinusoidal waveshape will not occur. Also, if the maximum magnitudes of the harmonics remain constant during the measurement, the standard can be used to calibrate a narrow-band phase-angle device.

The principal advantages of this phase-angle standard are its broad frequency range, its self-alignment features, its high accuracy, and its capability for use with sinusoidal or slightly harmonically distorted waveforms. If the measurement errors that have been identified are accounted for, there seems little reason why more sensitive galvanometers cannot be used and even higher accuracies realized in the measurement of the phase angle.

¹ A Standard for Accurate Phase-Angle Measurements at Audio Frequencies, by Winston W. Scott, Jr., NBS Tech. Note 347 (Oct. 14, 1966); available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, at a cost of 25 cents.

STANDARD REFERENCE MATERIALS



Standard Reference Materials are well-characterized materials disseminated by NBS to be used in calibrating and evaluating measuring instruments, methods, and systems, or to produce scientific data that can be referred readily to a common base. These materials are certified for chemical composition or for a particular physical or chemical property. They are used on-site in science and industry for calibrating the instruments and methods used for production and quality control of raw materials, chemicals, metals, ceramics, fuels, and radioactive nuclides in manufacturing processes and in research. This column regularly reports on the issuance of new and renewal Standard Reference Materials and on latest developments in the Standard Reference Materials Program.¹

NEW PLATINUM STANDARDS

Two new platinum standard reference materials, one of high purity and the other doped with impurity elements at the 5 to 10 ppm level, have recently been issued by the Bureau.

These standards, NBS No. 680, High-Purity Platinum, and NBS No. 681, Doped Platinum, are available in wire form 0.51 mm (0.020 inch) in diameter. Both of these are issued in two lengths, 10.2 cm (4 inches) and one meter (39.4 inches), designated L-1 and L-2, respectively. Standard No. 681 has been doped with a dozen impurity elements at the 5-10 ppm (by weight) concentration level. Both standards are certified for silver, gold, copper, iron, iridium, nickel, magnesium, oxygen, lead, palladium, rhodium, and zirconium. In general, values range from a fraction of a part per million in the high-purity platinum standard to about 10 parts per million in the doped standard. The L-1 standards cost \$35 each; the L-2 standards, \$185 each.²

At a conference held in 1960 between representatives of the platinum industry and the National Bureau of Standards, the foundation was laid for an extensive program to examine the effects of varying trace element concentrations on certain properties of high-purity platinum. One of the goals of the program was to study the relationships between the chemical composition and the electrical properties of platinum. It was planned that this program would result in two standards certified for chemical composition.

Both standards have been extensively tested and found satisfactory with respect to homogeneity. The following methods were used: optical emission spectrography; spark source mass spectrography; and electrical measurements, including electromotive force, temperature coefficient of

resistivity, and residual resistivity ratio. The recommended values of the concentrations of the various elements given on the provisional certificate were arrived at after analysis by one or more of the following methods: optical emission spectrography, spark-source mass spectrography (isotope dilution), polarography, spectrophotometry, nuclear activation analysis, and vacuum fusion. Cooperating with NBS in this extensive analytical program were: in the United States, J. Bishop and Co.; Sigmund Cohn; Engelhard Industries, Inc.; and RCA Laboratories; and, in the United Kingdom, Johnson, Matthey and Co., Ltd.

Historically, measurement systems at NBS from the very beginning have been fundamentally concerned with high-purity platinum. In 1922, the Bureau prepared a platinum wire material designated Pt 27. Without Pt 27, it is reported that the development of the present International Temperature Scale would not have been possible. Unfortunately, the supply of Pt 27 was totally exhausted and the need for a new material was becoming progressively acute in the 1950's. With respect to electrical measurements, another result of this cooperative program has been the provision of a platinum material which will be designated Pt 67, and which has essentially the same purity as Standard No. 680. Platinum 67 will be used at the Bureau as the national standard for the calibration of thermocouple wires.

NEW AND RENEWED SOLDER STANDARDS

Electrical and electronic industries utilize tremendous quantities of solder in making connections. Because a large part of soldering is now accomplished by automatic devices, control of the chemical composition of the solder has become increasingly important. Not only must the tin and lead content be carefully controlled, but also the minor elements such as antimony, bismuth, copper, arsenic, silver, and nickel. Improper chemical composition alters the temperature of the solder bath and is deleterious with regard to bonding of junctions and to adherence properties. Properly soldered junctions in space, defense, and oceanographic applications are of critical importance to successful mission accomplishment.

The first NBS solder standard, Standard No. 127, was issued in 1942 and had the nominal chemical composition of 35 weight percent tin and 65 percent lead. The first renewal, Standard No. 127a, was issued in 1951 and had the nominal chemical composition of 30 weight percent tin and 70 percent lead.

A renewal of NBS No. 127a, designated No. 127b, in the form of a metal powder, and a new solder standard, designated NBS No. 1131, in disk form, have been issued. Both of these new standards have a nominal chemical composition of 40 weight percent tin and 60 percent lead, which industry has indicated to be a more important chemical composition for present day needs than prior solder standards.

To obtain material of the highest possible homogeneity, 1500 pounds of alloy 40B (ASTM B32 specification) was converted by atomization to a fine metal powder. Of this powder 1000 pounds was retained for the chemical standard. The remaining 500 pounds was converted to 100 percent dense rods to serve as a standard material for optical emission and x-ray spectroscopic methods of analysis. Extensive testing revealed both lots of material to be of high homogeneity.

The solder standards are being made available initially with a provisional certificate of analysis covering the following elements: tin, antimony, bismuth, arsenic, copper, nickel, and silver.

The renewal standard, No. 127b, may be ordered in units of 150 grams of metal powder sized between 100 and 325 mesh. The price is \$28. The new standard, No. 1131, may be ordered in the form of disks 1 1/4 inches in diameter and 3/4 inch thick. The price is \$45.²

SUCROSE AND DEXTROSE STANDARDS

One of the more important classical instruments used by the sugar chemist is the polarimeter, which must be accurately calibrated. Renewals of two sugars, NBS Standard No. 17a, Sucrose, and NBS Standard No. 41a, Dextrose, have been prepared and certified for this purpose. These are successors to earlier issues, the stock of which has been exhausted.

Sugar is produced in greater tonnage than any other pure organic chemical. A significant portion of the annual production of raw sugar is imported and subject to a tariff based on the sucrose content, which is measured at the various ports of entry by means of accurately calibrated polarimeters.

It is mandatory that the tariffs levied on sugar by different customhouses be uniform. Before the Bureau issued standard sugars, each customhouse laboratory sent a weekly sample to NBS for analysis and results were circulated to all customhouses for comparison. With issuance of the NBS sucrose and dextrose standard reference materials, each customs laboratory is now able to calibrate its instruments daily and to check the equipment as frequently as necessary.

A very high grade, uniform sucrose with a very low concentration of ash and reducing substances was required for preparation of the standard. The sucrose is certified for its contents of moisture, ash, and reducing substances, as well as its specific rotation at 20°C at wave-

lengths of 5461 Å and 5892.5 Å. The sucrose from which No. 17a was prepared was supplied by the California and Hawaiian Sugar Refining Corp., Crockett, Calif. Standard No. 17a is sold in units weighing approximately 60 grams for \$21 a unit.² A certificate of analysis is supplied with each standard.

Dextrose, in addition to being certified for calibrating polarimeters, is used as an oxidation-reduction standard in the chemistry of carbohydrates and related materials. It is also used as a standard in clinical laboratories where the accurate determination of dextrose (glucose) is an increasingly important clinical and diagnostic tool.

Oxidation and reduction reactions are important in determining the structure and identification of sugars and sugar derivatives in industries that deal with carbohydrates, cellulose, corn products, and related substances. The dextrose standard provides a homogeneous material for calibrating the oxidation and reduction which will give end-effects physically similar to those of the carbohydrates under study.

The material from which Standard No. 41a was prepared was high-purity dextrose, with low ash and moisture content. The Pfanstiehl Laboratories, Inc., Chicago, Ill., supplied the dextrose for Standard No. 41a, which is sold in units weighing 70 grams for \$21 a unit.²

Chemical and physical testing of the sucrose and dextrose standard reference materials was performed by B. S. Carpenter, B. Coxon, R. A. Paulson, R. Schaffer, and B. F. West of the NBS Analytical Chemistry Division.

NEW PRICES FOR STANDARDS

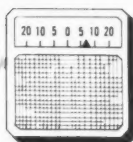
New prices for most standard reference materials went into effect December 29, 1967. These prices reflect a new method of determining production, certification, and distribution costs of the standards so that the users of the standards will pay the entire cost of this portion of the program.

Long-term support for the Standard Reference Materials Program, however, is still furnished by NBS through its appropriated research funds. These funds, which amount to three-fourths of total funds put into the program, support the Bureau's in-depth efforts in the basic measurement competences so vital to this work. The *Catalog and Price List* has been revised to reflect the new prices.¹

NOTE: The procedures for official announcements regarding Standard Reference Materials have been changed. For details, see *Standards and Calibration: Official Descriptions of Services and Standard Materials*, on page 64.

¹ Standard Reference Materials: Catalog and Price List of Standard Materials Issued by the National Bureau of Standards, NBS Misc. Publ. 260, 1968 Edition, for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 45 cents.

² Standards may be purchased from the NBS Office of Standard Reference Materials, Washington, D.C. 20234.



STANDARDS AND CALIBRATION

HIGH-FREQUENCY AND MICROWAVE CALIBRATION FEES RAISED

Calibration charges made to public customers by the Electronic Calibration Center at the NBS Radio Standards Laboratory were increased, effective the beginning of this year. Charges which were previously \$40 per hour (generally coaxial standards) are now \$45 per hour; charges which were previously \$45 per hour (generally waveguide standards) are now \$50 per hour. These increases in hourly rates were made necessary by the recent change in salaries and higher purchasing costs.

These changes in fees affect calibration services¹ in the high-frequency and microwave regions (30 kHz and up) which are performed by the Radio Standards Laboratory at Boulder, Colo.

NEW MEXICO RECEIVES NEW WEIGHTS AND MEASURES STANDARDS

M. W. Jensen, Chief of the NBS Office of Weights and Measures, presented a new set of weights and measures standards to New Mexico State Officials in a ceremony on December 8, at the University of New Mexico, University Park. With this presentation, six States* have now received new weights and measures standards in a program² to replace the standards of all 50 States at the rate of about ten per year.

OFFICIAL DESCRIPTIONS OF SERVICES AND STANDARD MATERIALS

Descriptions of NBS measurement services and standard reference materials, which hitherto became official upon publication in the Federal Register, are now made public via NBS Miscellaneous Publications 250 and 260, respectively. General statements concerning the overall services and reference materials will appear in the Federal Register, but details on specific services and materials will be given only in MP 250 and MP 260, and will be indicated in the Federal Register only through "incorporation by reference." Miscellaneous Publications 250 and 260 thus become, in effect, extensions of the Federal Register. The relevant general statements from the Federal Register will also be published in MP 250 and MP 260.

NBS Miscellaneous Publication 250, *Calibration and*

Test Services of the National Bureau of Standards, is available at \$1 per copy from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. It states NBS policies, describes NBS measurement services in detail, prescribes the procedures for obtaining such services, and lists the fees charged. As services are added or modified, supplements to MP 250 (known as "inserts") are sent to names on a mailing list of those wishing to be kept up-to-date. A postcard for requesting inclusion in the mailing list accompanies each copy of MP 250. It is planned to issue a new edition of MP 250, incorporating all changes to date, later this year.

NBS Miscellaneous Publication 260, *Standard Reference Materials Catalog and Price List of Standard Materials Issued by the National Bureau of Standards*, is sold at 45 cents per copy by the Superintendent of Documents (address above). It describes more than 600 different standard materials of metals, ores, ceramics, chemicals, and hydrocarbons now available from NBS, which are used to calibrate measurement systems and provide quantitative data that can be referred to a common base. Schedules of prices and quantities are given for all materials, as well as directions for ordering. Changes in availability or prices of various materials are given in quarterly insert sheets, sent to a mailing list similar to the one for MP 250.

STANDARD FREQUENCY AND TIME BROADCASTS

WWV —2.5, 5.0, 10.0, 15.0, 20.0, and 25.0 MHz
WWVH—2.5, 5.0, 10.0, and 15.0 MHz
WWVB—60 kHz

Radio stations WWV (Fort Collins, Colo.) and WWVH (Maui, Hawaii) broadcast signals that are kept in close agreement with the UT2 scale by making step adjustments of 100 ms as necessary. Each pulse indicates that the earth has rotated approximately 15 arcseconds about its axis since the previous one. Adjustments are made at 0000 UT on the first day of a month. *There will be no adjustment made on April 1, 1968.* The pulses occur at intervals that are longer than one second by 300 parts in 10^{10} due to an offset in carrier frequency coordinated by the Bureau International de l'Heure (BIH), Paris, France.

Radio station WWVB (Fort Collins, Colo.) broadcasts seconds pulses derived from the NBS Time Standard (NBS-III) with no offset. Step adjustments of 200 ms are made at 0000 UT on the first day of a month when necessary.

*California, Illinois, New Mexico, Ohio, Oregon, and Utah.

BH announces when such adjustments should be made in the scale to maintain the seconds pulses within about 100 ms of UT2. There will be no adjustment made on April 1, 1968.

BRIEF WWV SHUTDOWN

On January 24 last, NBS radio station WWV was shut

down on all frequencies for the purpose of installing an emergency power unit. The outage began at 1400 GMT and ended at 1805 GMT, when full service was restored.

¹ A description of these services, along with information about fees and general NBS policy relating to measurement services, is given in *Calibration and Test Services of the National Bureau of Standards*, NBS Misc. Publ. 250, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for \$1 per copy.

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Section B. Mathematical Sciences. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, \$2.75; foreign, \$3.50. Single copy, 75 cents.

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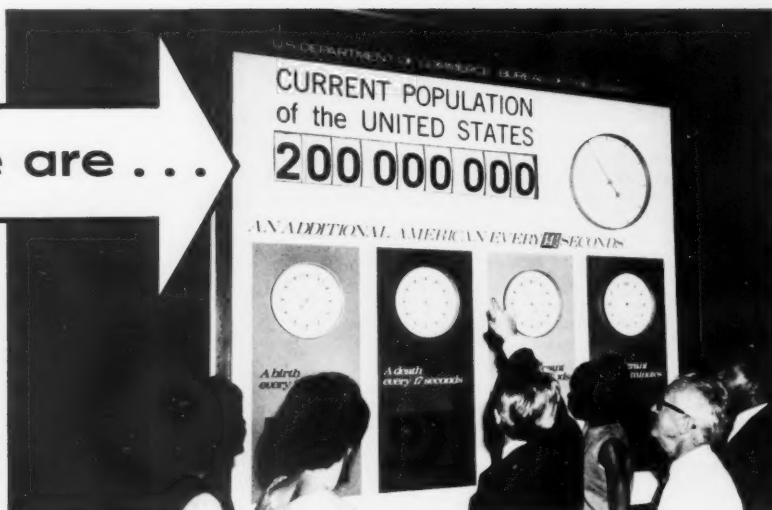
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